CITY OF MINNEAPOLIS WATER TREATMENT & DISTRIBUTION SERVICES

Revising Standard Method's Section 3125 Metals by ICPMS

Presented by Matthew Sullivan



Joint Task Group (JTG) established to review Standard Methods Section 3125 Metals

• Standard Methods Standard Operating Procedure-2023-2.0

• SOP for updating the source book on analytical SOP's

- Updated February 2023
- Like dark matter but you can see it if you look for it https://www.standardmethods.org/SOP

Ensures consensus methods retain inherent advantages SM 3125 is a consensus method not a government method

Protects against bias

• Ensures best practice

• Balance with respect to relevant and interested parties

Transparency

What is the Scope of the Charge?

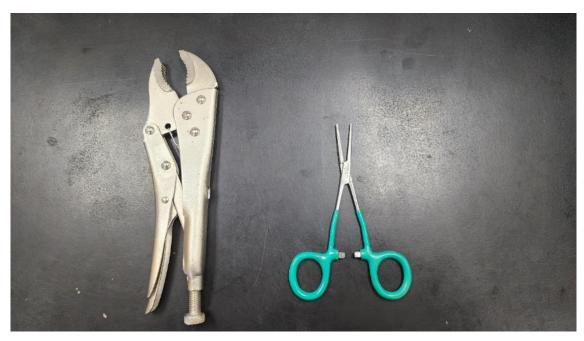
• A Systematic review of 3125 -editorial and technical

• Rare Earth Element (REE) correction equations

• Interference Removal Technology (IRT)

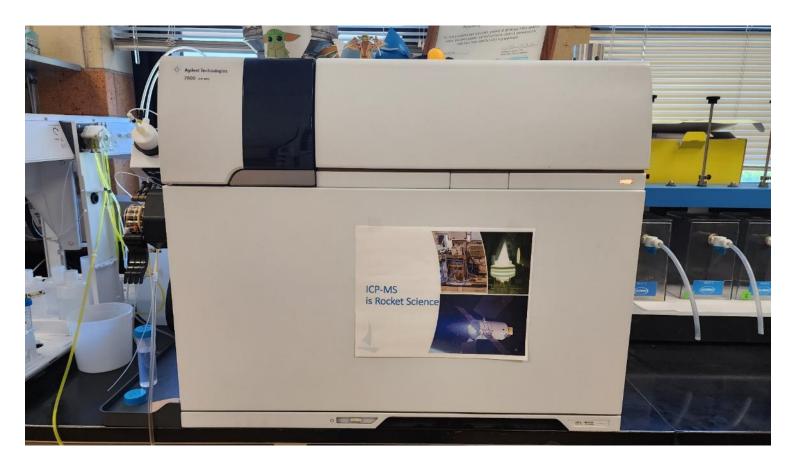
Why add Interference Removal Technology?

IRT allows for lower detection limits and increased confidence and defensibility



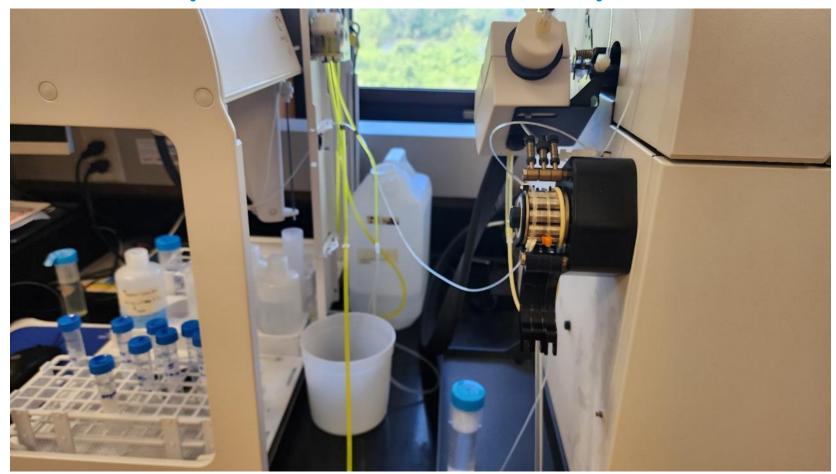
The right tool for the Job

Quick and depressing overview of ICPMS Instrumentation front



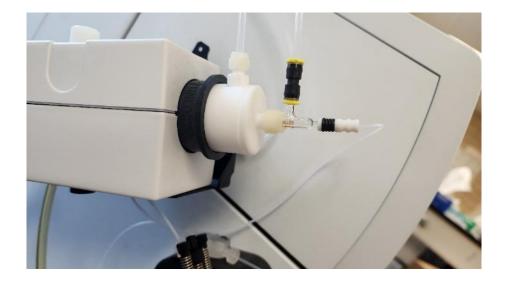
A magical breadbox full of pain and sorrow

Most service calls start and end with the sample introduction system



Peristaltic tubing shrivels if you look at it

Consistent nebulization is paramount



Everything physical and chemical Influences nebulization



Sample and preparation protocols are seldom trivial



There is no universal inorganic solvent

Joyous discussion of everything pre plasma that can go awry



Don't panic and bring a towel

Inside the torch temperatures rival the sun



- Molecules are atomized and ionized in torch
- Matrix lons react with each other forming polyatomic ions

Revenge of the matrix

- Mass to charge is what is detected at the detector m/z
- One unit mass resolution typical on modern mass spectrometers
- A polyatomic is indistinguishable from an analyte of same mass
- Arsenic weighs 10 septillionth of a gram more than ArCl (that is 30 zeroes)

A very real concern for many elements/matrices



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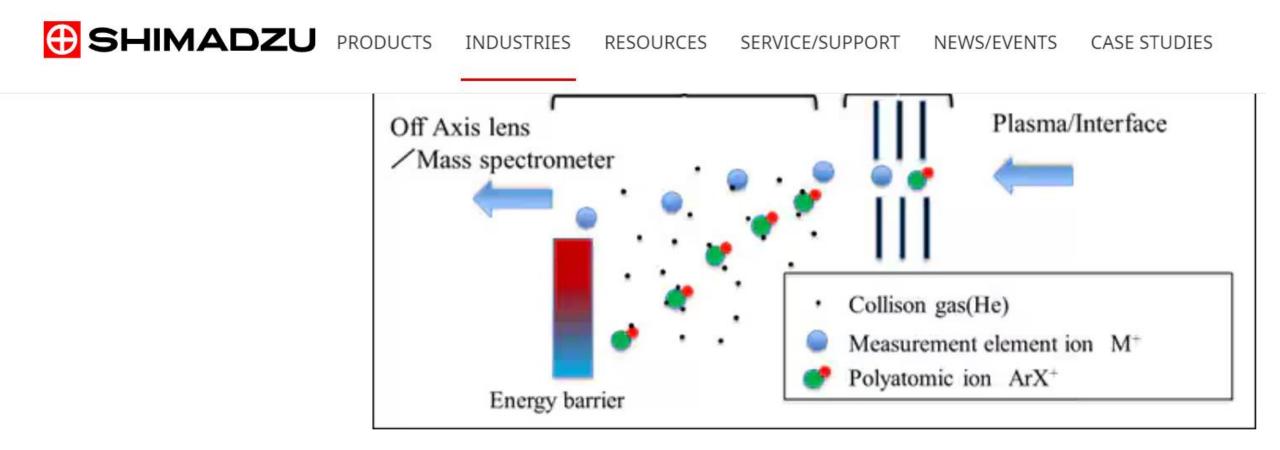
Other techniques only work so much

- Correction equations work great....to a point
- Optimization strategies lower but never eliminate polyatomic formation
- Time of Flight (TOF), X ray analyzers, and ICPOES are not substitutes



Brute Force Stock Photo -Square Peg In A Round Hole, Circle, Hole - iStock (istockphoto.com) Credit: ezoom

Interference removing technology to the rescue



https://www.ssi.shimadzu.com/industries/environment/icpms/index.html

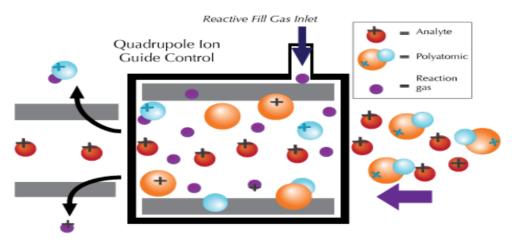
IRT is a combination term for both collision cell and reaction cell techniques

- Collision cell technology utilizes kinetic energy for resolution
- Reaction cell technology utilizes chemical reactions for resolution

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background levels resulting in dramatic improvements in detection limits and a reduction in biased resul for nearly the entire periodic table.

The DRC is a quadrupole enclosed within a reaction chamber (or cell) that is between the ion lens system and the analyzing quadrupole. A reactive gas such as NH₃ is introduced into the cell. The gas reacts with the ion beam through a number of ion-molecule reaction mechanisms, converting the interfering ions into species that will not interfere with the analyte. The analyte of interest, under the same conditions, remains stable and is able to proceed to the detector. This cleansing process is known as "chemical resolution".



https://brooksapplied.com/services/interference-reduction-technology/spectral-interference-removal-icp-drc-ms/

Not a perfect world



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But it is the best of all possible worlds

- Analytes also get removed in these pressurized parallel rod DC/AC cells
- Exponentially more interference than analyte removed
- Added complexity and moving parts
- Switching between IRT gas modes consumes precious seconds

Free the inorganic water laboratory



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What is the end goal for the JTG?

• A defining method for plasma-based instrumentation use in water

• interlaboratory study of interference check standard to validate IRT

• Validated according to SM part 1000 and SM SOP's

Report and/or published study on interlaboratory validation

• A yes vote by Standard Method's Joint Editorial Board (JEB)

Interference standard to validate IRT

• Other methods under review to include similar standard

• Discussion ongoing on number/nature of matrix/elements

• Important to empower the analytical chemist

Who is serving on JTG?

- Chair- Matthew Sullivan- Minneapolis Water Treatment and Distribution
- Part Coordinator Jennifer Calles- Mesa Water Resources Department
- William Lipps Nonvoting member-Shimadzu
- Sam Youssef- Fox River Water Reclamation District
- Dr. Brian Alexander Inorganic Ventures
- Joe Stussy- Phoenix Water
- Ben Wozniak Brooks Applied Speciation

EPA congruently updating EPA Method 200.8

- Adding IRT to EPA Method 200.8
- National Technology Transfer and Advancement Act (NTAA)
- NTAA instructs federal agencies to work on consensus methods
- Wherever possible and to avoid unique government methods

<u>The National Technology Transfer and Advancement Act (NTTAA)</u> directs Federal agencies to adopt voluntary consensus standards wherever possible (avoiding development of unique government standards) and establishes reporting requirements. NIST roles have the following objectives:

- Guide government agencies in conducting and reporting on standards and conformity assessment activities.
- Promote the use of standards developed by non-government organizations.
- Coordinate with Federal, state, and local agencies to foster greater reliance on voluntary consensus standards.
- Work through the Interagency Committee on Standards Policy (ICSP) to foster this reliance.

OMB Circular A-119, revised by the Office of Management and Budget (OMB) in January 2016, spells our the government strategy for standards development. It promotes agency participation on standards bodies, specifies reporting requirements on conformity assessment activities, and informs agencies of their statutory obligations related to standards setting.

https://www.nist.gov/standardsgov/what-we-do/federal-policy-standards/key-federal-directivesST

Meanwhile the EPA is updating 200.8

- A unique government method, made by the government
- NTAA should not be ignored but cautiously optimistic
- Extra encouragement to make SM 3125 a defining method

Why have we waited so many decades?

Government entities not wanting to stick necks out?

Lower detection limits for pollutants not welcome by all?

Water is inherently cautious?

The wild west of PFAS and plastic?

All the above?



Future goal for a future charge Inorganic Speciation



Photo taken by Austin Wilkes

The only place where speciation is not important is in regulation (and Standard Method's methods)

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Thank you for sticking around!

Questions?

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